

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460



OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

September 24, 2007

MEMORANDUM

SUBJECT: Review of "*Method Development for Measurement of Transferable Residues of Pyrethrin and Piperonyl Butoxide from Vinyl Flooring and Carpet by Using the Indoor Roller.*" (Project #: 98-024-PY01), MRID 461886-03, D336748.

FROM: Matthew Lloyd, Industrial Hygienist
Reregistration Branch 1
Health Effects Division (7509P)

A handwritten signature in black ink, appearing to read "m. lloyd".

THROUGH: Jeff Evans, Biologist
Chemistry and Exposure Branch
Health Effects Division (7509P)

A handwritten signature in black ink, appearing to read "j. evans".

TO: Cathryn O'Connell, Chemical Review Manager
Reregistration Branch 2
Special Review and Registration Division (7508P)

PC Code: 067501, 069001

Attached is a review of a study conducted to develop a method for measuring transferable residue of Pyrethrin (PY) and Piperonyl Butoxide (PBO) from a homogeneous floor covering (carpet or vinyl) to an indoor roller. The study was submitted by the Pyrethrin/Piperonyl Butoxide/MGK-264/Deltamethrin Non-Dietary Exposure Task Force. As this study is a method development study, it did not undergo the typical primary and secondary review process. The following serves as a complete review of this method development study and its findings.

Summary

The objective of this study was to provide method development for measurement of transferability of residues using the indoor roller. The method was tested on both carpet and vinyl flooring with two active ingredients, pyrethrins (PY) and piperonyl butoxide (PBO).

Specific study objectives included:

1. Test the utility of the method and determine necessary modifications to the method;
2. Verify the detection limits needed to obtain measureable residues of the two active ingredients under the test conditions;
3. Provide a range finding study for transfer efficiency using the indoor roller method.

A prototype total aerosol release indoor fogger developed by MGK (0.78% PY and 1.5% PBO) was used as the test substance. The test substance was applied using a sprayboom apparatus in the center of a 16 ft. x 16 ft. x 8 ft. test rooms (Simulated Residential Rooms or SRRs). The desired deposition rate of the test material onto the vinyl flooring was $3.96 \mu\text{g}/\text{cm}^2$ for PY and $7.87 \mu\text{g}/\text{cm}^2$ for PBO.

The study focused on the measurement of transferred residues onto the test surface using the indoor roller. Percalé was used as the test surface material and alpha-cellulose was used as the test surface material for the residue coupons.

The overall field and laboratory recoveries were in acceptable range. Results are shown in the table below.

Fortified Field and Laboratory Controls – Mean Recoveries & Standard Deviations

Sample Type	PYI/PY	PBO
Laboratory Fortified Percalé Controls	120±13%	130±21%
Field Fortified Alpha-Cellulose Controls	101±9.1%	85.3±6.3%
Field Fortified Percalé Controls	101±6.6%	71.2±15%
Laboratory Fortified Alpha-Cellulose Controls	91.7±12%	88.9±8.9%

Bolded Field Fortification Recoveries were reported in the study as less than 90% and require correction per HED policy.

Field fortification samples were prepared and spiked at 1X, 2X, and at least 5X the LOQ. Field fortification samples were prepared in triplicate and exposed for the 3 hour post-application deposition period. The validated LOQs for percalé are $0.0005 \mu\text{g}/\text{cm}^2$ for PY and $0.0003 \mu\text{g}/\text{cm}^2$ for PBO. The validated LOQs for the alpha cellulose coupons are $0.009 \mu\text{g}/\text{cm}^2$ for PY and $0.040 \mu\text{g}/\text{cm}^2$ for PBO.

There were 2 PYI fortifications for alpha-cellulose coupons, 0.353 and 212 $\mu\text{g}/\text{sample}$, respectively. There were 2 PBO fortifications for alpha-cellulose coupons, 2.79 and 466 $\mu\text{g}/\text{sample}$ for alpha-cellulose coupons. Recoveries were close for the two field fortification levels for PY. The PBO recoveries were generally below 90% and corrected according to HED policy.

An initial set of field fortified percale coupons were excluded due to unexplained extremely high recovery values. There were 2 PYI fortifications in the revised percale coupon set, 0.0353 and 0.176 $\mu\text{g}/\text{sample}$. There were 2 PBO fortifications for percale coupons, 0.0372 and 0.186 μg for alpha-cellulose coupons. Recoveries for the two PY field fortification levels were close. The PBO recoveries for both the low and high field fortifications were all below 90% and corrected according to HED policy.

Background deposition was measured in the SRR two days prior to application with similar ambient conditions to the actual application day. PY and PBO on background coupons (n=2) were below detection limits.

Coupons and dosimeters were placed on the platforms in the test room. After the removal of the deposition coupons from the deposition surface on the platforms, the indoor roller assembly was carefully fitted to the platforms on each test lane. After use of the roller, the assembly was washed and refitted to the next lane.

The profiles of PY and PBO on the test platforms are shown below.

CARPET				VINYL			
3.42 5.82	7.10 11.76	6.19 10.36	5.47 9.62	5.94 9.70	3.85 6.39	2.89 4.87	4.16 6.66
Pyrethrins (Py) Deposition Coupon = 5.631 $\mu\text{g}/\text{cm}^2$ (N = 12) Indoor Roller = 0.203 $\mu\text{g}/\text{cm}^2$ (N = 2) Fraction Transferred = 0.036				Pyrethrins (Py) Deposition Coupon = 5.543 $\mu\text{g}/\text{cm}^2$ (N = 12) Indoor Roller = 0.130 $\mu\text{g}/\text{cm}^2$ (N = 2) Fraction Transferred = 0.023			
4.41 7.56	6.42 10.69	5.82 9.78	6.34 10.73	6.13 9.76	4.60 7.53	6.63 11.12	6.26 10.48
Piperonyl Butoxide (PBO) Deposition Coupon = 9.484 $\mu\text{g}/\text{cm}^2$ (N=12) Indoor Roller = 0.453 $\mu\text{g}/\text{cm}^2$ (N = 2) Fraction Transferred = 0.047				Piperonyl Butoxide (PBO) Deposition Coupon = 9.132 $\mu\text{g}/\text{cm}^2$ (N=12) Indoor Roller = 0.183 $\mu\text{g}/\text{cm}^2$ (N = 2) Fraction Transferred = 0.020			
4.07 6.93	6.04 10.05	6.35 10.62	5.94 9.75	7.29 12.11	6.26 10.29	5.66 9.38	6.85 11.30
Note: The small boxes, with a solid outline, represent deposition coupons as they were positioned on platforms. In these boxes, the upper value is for Py and the lower one (in <i>italics</i>) is for PBO, both in $\mu\text{g}/\text{cm}^2$. The larger boxes, with a dashed outline, are regions where either carpet or vinyl were placed.							

Relevant SOPs/Validation in Project: 98-024-PY01 (MRID 46188603)

Scenario	SOP/Validation
Simulated Residential Room (SRR) Set-up	Toxcon SOP E-025: <i>Preparation of Test Rooms Prior to an Experiment</i>
Spray Boom Operation	Toxcon Report 98-033-PY01: <i>Validation Plan for the Whitmyre Application System for Pesticides</i>
SRR Cleaning/Decontamination	Toxcon SOP M-027
Deposition Coupon QA	Toxcon Method M-015
Percule Dosimeter QA	Toxcon Method M-018
GC/MS procedure	XAM-48.V1

Study Conclusions

The method using a spray boom and an indoor roller generated transferable residue fractions for carpet and vinyl flooring. The targeted deposition rate for the deposition coupons were 3.96 $\mu\text{g}/\text{cm}^2$ for PY and 7.87 $\mu\text{g}/\text{cm}^2$ for PBO. The overall deposition rate for all coupons was determined to be 5.59 ± 1.21 $\mu\text{g}/\text{cm}^2$ for PY and 9.30 ± 1.99 $\mu\text{g}/\text{cm}^2$ for PBO. The target deposition rate was exceeded for PY by 41% and for PBO by 18%. The performance of the spray boom was close to the protocol specified limits of 20% coefficient of variation (COV) (20.6% for PY and 19.8% for PBO).

PY and PBO Residue Data

Sample	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$	Sample	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$
1AV	6.85	12.6	1AC	5.94	10.9
1BV	5.66	10.5	1BC	6.35	11.9
1CV	6.26	11.5	1CC	6.04	11.2
1DV	7.28	13.5	1DC	4.07	7.8
3AV	6.26	11.7	3AC	6.33	12.0
3BV	6.63	12.4	3BC	5.82	10.9
3CV	4.60	8.4	3CC	6.41	12.0
3DV	6.12	10.9	3DC	4.41	8.5
5AV	4.15	7.5	5AC	5.47	10.8
5BV	2.89	5.5	5BC	6.19	11.6
5CV	3.84	7.1	5CC	7.10	13.2
5DV	5.95	10.9	5DC	3.43	6.5

A – Corrected for field recoveries of >90% per HED policy.

The study authors reported that the operation of the indoor roller assembly was smooth but slow to use. Inter-application clean-up and re-assembly times were higher than normal. Additionally, the study authors noted that the operation of the roller in the base plate assembly does not fully expose the entire dosimeter exposure surface.

PY and PBO Residue Statistics on Carpet

Statistic	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$
Test Surface	Coupons	Coupons	Dosimeter	Dosimeter
Count, n	12	12	2	2
Mean, μ	5.63	10.6	0.203	0.613
Standard Deviation	1.1	2.0	0.0006	0.005
Coefficient of Variation	0.20	0.19	--	--

A – Corrected for field recoveries of >90% per HED policy.

PY and PBO Residue Statistics on Vinyl

Statistic	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$	PY, $\mu\text{g}/\text{cm}^2$	PBO ^A , $\mu\text{g}/\text{cm}^2$
Test Surface	Coupons	Coupons	Dosimeter	Dosimeter
Count, n	12	12	2	2
Mean, μ	5.54	10.22	0.130	0.250
Standard Deviation	1.36	2.5	0.054	0.096
Coefficient of Variation	0.24	0.25	--	--

A – Corrected for field recoveries of >90% per HED policy.

The average fraction transferred from the carpet to the percale dosimeter was 3.6% for PY and 5.7% for PBO. The average fraction transferred from the vinyl to the percale using the indoor roller was 2.3% for PY and 2.4% for PBO.